Biaxial flexural strength and Weibull characteristics of adhesively luted hybrid and reinforced CAD/CAM materials to dentin: effect of self-etching ceramic primer versus hydrofluoric acid etching

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Abstract: This study evaluated the influence of the surface treatment and aging on the biaxial flexural strength of ceramic materials cemented to a dentin analogue. One hundred twenty disc-shaped specimens were allocated into 12 groups considering three study factors: ceramic material (lithium disilicate, leucite-based ceramic and hybrid ceramic), surface treatment (10% hydrofluoric acid etching + silane or self-etching glass-ceramic primer) and Aging (with 10,000 thermocycles of 5–37–55 ℃ or without). A tri-layer assembly was designed to mimic a cemented restoration (Variolink N) into a dentin analogue. All samples were submitted to the biaxial flexural strength assay. The flexural strength in MPa was calculated using the finite element method for each sample considering thickness, material properties, and the load to fracture during the in vitro test. Fractographic analysis was also performed. The data was evaluated using three-way ANOVA and Tukey test (α = 5%). ANOVA showed influence for the Material*Treatment*Aging interaction on the flexural strength (p = 0.011). The highest strength was calculated for lithium disilicate ceramic + self-etching ceramic primer without aging (499 ± 17 MPa)A and the lowest value for hybrid ceramic material + acid etching with aging (424 ± 48 MPa)E. According to the Weibull modulus, the most predictable strength was calculated for lithium disilicate + acid etching after aging. Acid etching or self-etching ceramic primer promotes similar immediate biaxial flexural strength for each evaluated ceramic. In the long-term, superior strength was observed using acid etching for lithium disilicate and the self-etching ceramic primer for the hybrid ceramic while no difference was observed for leucite-based ceramic.

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Biaxial flexural strength and Weibull characteristics of adhesively luted hybrid and reinforced CAD/CAM materials to dentin: Effect of self-etching ceramic primer versus hydrofluoric acid etching

Running title: Biaxial flexural strength of CAD/CAM materials

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ABSTRACT

This study evaluated the influence of the surface treatment and aging on the biaxial flexural strength of ceramic materials cemented to a dentin analogue. 120 disc-shaped specimens were allocated into 12 groups considering three study factors: ceramic material (Lithium disilicate, Leucite-based ceramic and Hybrid ceramic), surface treatment (10% Hydrofluoric acid etching + silane or Self-etching glass-ceramic primer) and Aging (with 10,000 thermocycles of 5-37-55°C or without). A tri-layer assembly was designed to mimic a cemented restoration (Variolink N) into a dentin analogue. All samples were submitted to the biaxial flexural strength assay. The flexural strength in MPa was calculated using the finite element method for each sample considering thickness, material properties and the load to fracture during the *in vitro* test. Fractographic analysis was also performed. The data was evaluated using three-way ANOVA and Tukey test (α=5%). ANOVA showed influence for the Material* Treatment*Aging interaction on the flexural strength (p=0.011). The highest strength was calculated for lithium disilicate ceramic + self-etching ceramic primer without aging (499 ± 17 MPa)^{A} and the lowest value for hybrid ceramic material+ acid etching with aging (424 ± 48 MPa)^{E}. According to the Weibull modulus, the most predicable strength was calculated for lithium disilicate + acid etching after aging. Acid etching or self-etching ceramic primer promote similar immediate biaxial flexural strength for each evaluated ceramic. In the long-term, superior strength was observed using acid etching for lithium disilicate and the self-etching ceramic primer for the hybrid ceramic while no difference was observed for leucite-based ceramic.

Keywords: Adhesion; CAD/CAM ceramics; finite element analysis; self-etching ceramic primer; surface treatment.

CLINICAL IMPLICATIONS

Some protocols combining the CAD/CAM ceramic material and the surface treatment could present suitable and stable flexural strength.

INTRODUCTION

The use of indirect restorations allows great coronal destruction to be rehabilitated with adequate aesthetics and resistance, recovering the masticatory function and protecting dental remnant.[1] Among indirect restoration methods, the CAD/CAM (Computer aided design/Computer aided manufacturing) stands out because it enables obtaining homogeneous restorations with a lower number of structural defects,[2] lower residual stress and shorter laboratory time.[3] The promising features of the CAD/CAM milling method led to the development of a variety of restorative materials that can be used as raw material. Thus, the clinician finds difficulties to determine the best restorative material for their need since several of these materials have adequate aesthetics[2] and acceptable success ratios.[4-6]

Ceramic materials that have a glass matrix in their composition require surface treatments prior to the adhesive cementation.[7] The hydrofluoric acid etching protocol is commonly used because it modifies the ceramic material creating mechanical retention[8] and providing a reactive surface to interact with the molecules of the resinous cement.[9] All ceramic material for CAD/CAM technology requires a surface treatment so that adequate bond strength to the substrate is achieved. Different materials have different compositions according to the amount of glass matrix. Each surface consequently needs a specific time for the acid conditioning to be effective.[2,7,9] The conditioning time is an important clinical step to avoid the formation of deep defects that could result in a decrease in bond strength.[10] Which reinforces the importance to perform correctly the cementation procedure that also fill the surface defects with the resin cement and increases the restoration survival.[11] Thus, studying an adhesively-cemented restoration is a more realistic approximation of the conditions found in the oral environment.[11]

After treatment with hydrofluoric acid, the conditioned surface must be cleaned, and then a silane agent must be applied.[7,12-18] The inorganic molecules of the restorative material require a bonding agent that facilitates the chemical bonds with the organic molecules of the resin cement. Thus, the function of the silane agent is to significantly improve adhesion between the ceramic and cement.[9]

Therefore, a self-etching glass-ceramic primer was developed to be applied on glass-ceramic materials in order to reduce the number of clinical steps.[7] This primer acts etching and silanizing the surface of the ceramic material through a single application step. Self-etching glass-ceramic primer has a single exposure time, independent of the chosen material, which could standardize the conditioning protocol facilitating the cementing procedure of different restorations.[7,19,20] As this material is indicated for a few types of ceramic materials, it is necessary to know its effect on materials with different compositions, as well as to verify the performance of surface conditioning with self-etching glass-ceramic primer in the long-term. There is no consensus regarding the superiority of hydrofluoric acid or self-etching glass-ceramic primer.[8,12-18,21,22] Thus, the goal of this study was to evaluate the influence of the surface treatment on the immediate and long-term biaxial flexural strength of three ceramic materials cemented to a dentin analogue (Epoxydplatte; Carbotec GmbH & Co. KG, Germany). The hypothesis was that there would be no difference between surface treatments on the biaxial flexural strength regardless the ceramic material and aging.

MATERIALS AND METHODS

Specimen preparation

Blocks of each ceramic material for CAD/CAM (Lithium disilicate glass-ceramic, e.max CAD, Ivoclar Vivadent; Hybrid ceramic, VITA Enamic, VITA Zahnfabrik; and Leucite-based glass-ceramic, Empress CAD, Ivoclar Vivadent,) were rounded in an automatic orbital sander (Ecomet 250, Buehler) using wet sandpaper with grain size #600. Next, the cylindrical rollers that resulted were cut using a precision cutting machine (Isomet 1000, Buehler) into discs under constant water irrigation (40 discs/material). All specimens were then finished in the orbital sander (Ecomet Polisher, Buehler LTD, USA) with an applied force of 30 N at a speed of 450 rpm for 5 min with different grits of silicon carbide grinding paper (#120, 240, 320, 600 grit sandpaper) under running water to assure surface smoothness and parallelism before the biaxial flexural strength test. The specimens' final dimensions were 10 mm diameter and 1.2 mm thickness. Afterwards, all samples were cleaned with isopropyl alcohol in an ultrasonic bath (5 min). The lithium disilicate discs were crystallized in a specific oven (Programat P700, Ivoclar Vivadent, Schaan, Liechtenstein) according to the manufacturer's instructions. The materials respective manufacturers and compositions are summarized in table 1.

The epoxy resin material that served as bonding substrate (Protec, São Paulo, Brazil) was cut in established standardized thickness (2.5 mm) and diameter (10 mm). It was then polished with #600 and #1200 grit silicon carbide papers to assume a final thickness of 2.3 mm. The epoxy resin discs were cleaned with isopropyl alcohol in an ultrasonic bath (5 min). A setup was designed to reproduce an occlusal restoration for a posterior tooth following the methodology by Guilardi et al. (2019).[23] A diameter of 10 mm was used to mimic the average area of a first molar.[24] The cemented assembly had a final thickness of 3.5 mm (ceramic thickness of 1.2 mm + epoxy resin thickness of 2.3 mm) to achieve equivalent thickness to the average thickness from the roof of the pulp chamber to the occlusal surface.[25]

Study design

The ceramic and epoxy resin discs were randomly assigned into 6 groups according to the design shown in Table 2 (n=10): ceramic material (lithium disilicate, leucite-based ceramic and hybrid ceramic), surface treatment (hydrofluoric acid etching or self-etching glass-ceramic primer) and long-term aging (with or without). A flowchart of the allocation of experimental groups are present as figure 1.

Surface treatments and cementation

The detailed ceramic surface treatments are summarized in Table 2. While, for the bonding substrate, the cementation surface was etched with 10% hydrofluoric acid (Condac porcelana, FGM) for 60 s, followed by ultrasonic cleaning in distilled water (5 min), and then application of Multilink Primer A plus Multilink Primer B mixture of Multilink N resin cement system (Ivoclar Vivadent), using a microbrush under vigorous movements for 15 s and gentle air-drying for excess removal.

Dual-cure resin cement (Multilink N, Ivoclar Vivadent) was manipulated as recommended by the manufacturer and applied on the center of the treated ceramic surfaces. The discs were joined with a standard load of 7.5 N on the occlusal surface of the ceramic, promoting uniform cement spreading. The excess cement was removed using a microbrush and light curing (high intensity of 1000 mW/cm²; wavelength ranging from 395 to 480 nm - Valo, Ultradent Products) was performed for 20 s on the occlusal surface of the ceramic, followed by 10 s in four points of the bonded interface (0°, 90°, 180°, 270°). After 48 h of immersion in distilled water, the samples were divided in two subgroups: without and with aging (Fig. 1 and Table 2).

Aging protocol

Half of the samples was submitted to thermalcycling (5-37-55°C/10,000 cycles) to investigate the bond stability over a long-term and its influence on the biaxial flexural strength. There is no consensus according the best accelerated aging procedure.[26] However, 10,000 thermocycles are suggested as corresponding to 1 year of *in vivo* environment.[27] Based on this information and for an optimized clinical situation, the thermal sequence used included the reference *in vivo* resting temperature of 37 °C.[28]

Biaxial flexural strength test

All samples were tested positioned in a stainless steel base (mounting bracket) with their flat surface aligned perpendicular to the source of the applied load in a universal testing machine (DL1000, EMIC) for the biaxial flexural strength test. The load was applied to the ceramic disc external surface by a unidirectional vertical device (ϕ=3mm, 1000 kgf load cell, under water) with a crosshead speed of 0.01 mm/min until failure occurred. The fracture load to start the failure (N) was computer recorded. To record the failure, this study followed a previously published methodology that interrupted the test when the initial cracking sound was detected.[29]

Calculation of the biaxial flexural strength at fracture

The biaxial flexural strength at fracture correspond to the maximum tensile stress in the center of the specimen.[30,31] For this study, the maximum center tensile stress was calculated using the finite element analysis (FEA) according to the ceramic material and aging protocol. In the absence of a computer aided engineering software, the manually calculation can be performed using Hsueh et al. (2006, 2009)[30,32] formula:

$$\sigma i = \frac{Ei(z-z*)M}{(1-vi)(1+vave)D*} \quad \text{(For } i = 1 \text{ to n)}$$

Where E is the Young's modulus of the layer, M is the biaxial bending moment per unit length, z* is the position of neutral plane, D* is the flexural rigidity and v_{ave} is the Poisson ratio average.[33] The flexural rigidity, and the average Poisson ratio of the multilayered disc should be calculated according to Huang et al. (2011) depending of the layer numbers and properties.[33] Nevertheless, for trilayered discs, the finite element methods result for piston-on-three-ball tests, provide good estimation of the maximum tensile stress at the center of the discs.[30-33] For this approach, the three-dimensional (3D) model of the *in vitro* assay was designed with the same dimensions as the testing specimens containing the ceramic disc, the cement layer and dentin analogue.[11,30,34] For the correct modeling, one cemented sample was embedded into acrylic resin and cut in two parts with a low-speed diamond saw with water-cooling to produce a realistic modeling of the *in vitro* specimen in the computational simulation. Each slice was examined and photographed by stereomicroscopy under 7.5x magnification. The photographies were used as background for the 3D geometries modeling using a computer-aided software (Rhinoceros, version 5.0 SR8, McNeel North America). The final geometries (Fig. 2) were imported to the analysis software (ANSYS 17.2, ANSYS Inc.) in STandard for the Exchange of Product (STEP) data format. Tetrahedral elements formed the mesh after a convergence test (10%) and each material's mechanical properties were assigned as having isotropic behavior (having identical property values in all directions).[35] The Young's modulus and Poisson ratio used for the calculation were based in previous papers and the reference list can be found in Dal Piva et al., 2018.[36] The data of each *in vitro* fractured sample (N) was used to perform an individual simulation (Fig. 3) and obtain a specific value in Megapascal.[30,32] The flexural strength data was exported and submitted to a statistical analysis.

Failure analysis

The tested samples (radial cracks) were perpendicularly cut in two halves[23] and inspected using a stereomicroscope (Stereo Discovery V20, Zeiss) (Fig. 4) and a scanning electron microscopy (SEM - Inspect S 50 – FEI Company) to determine the failure origin and direction of crack propagation (Fig. 5). For SEM, the samples were perpendicularly cut into two parts for comparison with the three-dimensional model. The specimens were sputter-coated with gold for 130 s at 15 mA, creating a 30-nm-thick layer and examined under different standard magnifications operated at 20 KV using secondary electron detection by a single operator.

Statistical analysis

After the data normality verification using the Kolmogorov-Smirnov test, the biaxial flexural strength data were analyzed by descriptive statistics and three-way ANOVA followed by multiple comparison post-hoc Tukey test. α=5% was considered statistically significant in all tests. The data obtained in the biaxial flexural test were subjected to Weibull analysis to identify the Weibull moduli and characteristic strength.

RESULTS

Three-way ANOVA showed that material and aging factors influenced the biaxial flexural strength values. Lithium disilicate showed higher biaxial flexural strength $(482.9 \pm 39.6)^{A}$ than leucite based-ceramic $(440.5 \pm 28.5)^{B}$ and hybrid ceramic $(455.2 \pm 55.7)^{B}$. Non-aged groups $(472.0 \pm 45.3)^{A}$ showed superior flexural strength than aged groups $(447.1 \pm 43.4)^{B}$. Material* Treatment*Aging interaction influenced on the mean biaxial flexural strength among the groups (Table 3). According to Tukey test lithium disilicate etched with self-etching ceramic primer immediate after the cementation showed the highest flexural strength while acid etched hybrid ceramic showed the lowest mean value. Both surface treatments for hybrid ceramic and leucite-based ceramic showed stable flexural strength values. While, for lithium disilicate, the flexural strength was not stable in the long-term when the self-etching glass-ceramic primer was used. Table 3 shows that the most suitable treatment (higher and stable mean values) for hybrid ceramic was the self-etching glass-ceramic primer, while for the lithium silicate was the acid etching and no difference was observed for leucite-based ceramic.

Fractographic analysis using a stereomicroscope under different illuminations (Fig. 4) showed that the failure origin was located on the ceramic cementation surface (tensile side). According to SEM micrographic images, all failures started from the cementation surface as radial cracks (Fig. 5). It was possible to observe fractures starting from defects located on the tensile surface of the ceramic (i.e. on the cementation surface).

The graph (Fig. 6) has the statistical Weibull numbers with the aligned distribution of values. The steeper the line, the higher the modulus and the greater the homogeneity of the setup. The highest Weibull modulus was calculated for lithium disilicate with acid etching after aging, what implies that this group present the most predicable behavior.

DISCUSSION

The goal of this study was to evaluate the influence of the surface treatment on the immediate and long-term biaxial flexural strength of three ceramic materials cemented to a dentin analogue. Results showed that material and aging influenced the biaxial flexural strength, rejecting the null hypothesis. The main finding of the current study was that only the lithium disilicate ceramic treated with the self-etching glass-ceramic primer demonstrated a significant decrease on the flexural strength value stability (significant strength reduction after thermal aging). Instead, stable mean values were found for all the other groups regardless of the surface treatment.

The biaxial flexural strength data in MPa were obtained performing a calculation for each sample based on the value of load to fracture during the *in vitro* test. This approach has been already performed by previous studies to validate the formulas for manual calculation.[30,31,37] According to the theory of maximum center stress, there is an acceptable similarity between both manual calculus and computational (*in silico*) method.[30-32]

A previous study observed that the adhesion between resin cement and CAD/CAM materials was influenced by the surface conditioning method, aging and material.[38] This affirmation assists the explanation of the results observed in this study; once the interaction of these factors was significant to influence the biaxial flexural strength of cemented restorations.

The leucite-based ceramic (SiO_2, Al_2O_3, K_2O, Na_2O, other oxides and pigments) and the lithium disilicate ceramic (SiO_2, Li_2O, K_2O, P_2O_5, ZrO_2, ZnO, Al_2O_3, MgO and colouring oxides) are rich in silica, being considered as acid-sensitive ceramics. The protocol used for hydrofluoric acid etching consists of 20 s for lithium disilicate and 60 s for the leucite-based ceramic.[7] Considering hydrofluoric acid etching on the hybrid ceramic (SiO_2, Al_2O_3, Na_2O, K_2O, B_2O_3, ZrO_2, CaO, TiO_2, Urethane dimethacrylate and triethylene glycol dimethacrylate) there was no significant difference between using 10% acid etching for 20 or 60 s.[39] It is in the glass-matrix structure that the etching with hydrofluoric acid will form micro retentions, modifying the surface topography and forming valleys and microscopic peaks responsible for the stress accumulation and crack propagation.[39,40] These defects in the ceramic surface become less critical to the material's strength when performing an adhesive procedure due to the filling with resinous cement.[2,11]

An alternative to the etching with the hydrofluoric acid is the use of a self-etching agent. This material enables performing acid etching and silanization in a single step, reaching adhesive bond strength similar to the values promoted by the hydrofluoric acid conditioning in glass-ceramics.[7,12,15,16,20,21] However, these studies used methodologies that exclusively evaluate the bond strength, such as shear/microshear[12,13,14,17,18,41] or microtensile[7,15,16,19] assays. Since most of the studies with shear test suggest more promising results for the acid conditioning whereas studies using microtensile show similarity in the average values; this study used an alternative method to evaluate the influence of the thermal aging factor on the cementation of ceramics treated under different methods. The present study compared both surface treatments in a simplified prosthesis cemented on an analogous material to dentin.[11,20] The configuration of the specimen cemented on a substrate has been validated, since it corresponds to the most realistic restoration condition found in the oral environment.[11,42]

All the ceramic materials studied in this study have been reported as susceptible materials to receive both hydrofluoric acid and self-etching glass-ceramic primer.[7,20,39] The etching protocol with 10% hydrofluoric acid for 20s is suggested for hybrid ceramic as enough to roughen the surface and preserve most of the silica content.[39] However, the effect of thermal degradation on the adhesive interface using the load-to-fracture of these ceramics has not been evaluated yet.

The use of finite element method has previously been described as a complementary method to laboratory findings.[11,34] The decrease in the fracture loading values for the aged groups reduced the critical stress during failure, showing a reduced strength. There was no statistical difference for surface treatments in this investigation, but the interaction of all factors was significant. Thus, each material showed an immediate result after the cementation that aging may or may not influence depending on the proposed surface treatment.

Lithium disilicate treated with the self-etching glass-ceramic primer had the highest biaxial flexural strength immediately after cementation (± 500 MPa). However, it was the unique condition that showed a significant reduction (10.7%) in the flexural strength after aging. Lopes et al. (2018)[17] compared lithium disilicate bond strength to a resin cement using a self-etching glass-ceramic primer or a hydrofluoric acid using microshear assay. The authors observed that the self-etching glass-ceramic primer promotes lower bond strength values than the hydrofluoric acid after 48 h. This finding was different from that observed by other studies[7,12,19,22] that observed no difference between both treatments in the short-term.

No difference was observed for the lithium dilicate bond strength[7,15,16,21] for the long-term evaluation. However, the self-etching glass-ceramic primer showed a decrease in the mean values for lithium disilicate after 10,000 thermalcycles. This result could be justified by the less etching depth promoted by the self-etching primer on the lithium disilicate surface[39] which could be insufficient to promote durable mean value of flexural strength in the long-term (Table 2). This slighter surface alteration is suggested to reduce the number of flaws on the ceramic cementation surface, increasing the fatigue failure load for lithium dissilicate.[20] The authors stair case fatigued lithium dissilicate discs in the same specimen configuration used in this study; and observed that the self-etching primer promotes the best fatigue behavior compared to other surface treatments.

On the other hand, a decrease in the flexural strength for the leucite-based ceramic was observed for both surface treatments, but none of them was statistically different from the mean values without aging. This is in accordance with a previous study that concluded that both treatments could be a conditioning options for leucite-based ceramic without difference of bond strength.[41] The mean value of failure stress of the hybrid ceramic treated with the self-etching glass-ceramic primer prior to aging reached statistically similar values to the lithium disilicate. In contrast, the use of hydrofluoric acid on the hybrid ceramic reached similar values as the leucite-based ceramics. Neither of the surface treatments after aging significantly reduced the flexural strength for the hybrid ceramics without aging. However, in comparing both treatments after aging, the self-etching glass-ceramic primer showed a promising performance for the hybrid ceramic, different from other study[22] that found similar results for both surface treatments on the hybrid ceramic bond strength.

It is important to mention that the performed experiment represents the maximum load to start cracking on a cemented specimen. Thus, the mechanical test was stopped shortly after the first failure sound.[29] Therefore, it was possible to observe that all discs presented failure origin at the adhesive interface without debonding from the ceramic part for all samples. Samples that catastrophically failed were replaced with new specimens.

Ceramic materials do fail under tensile stresses.[43] In the present study, each sample was subjected to stereomicroscope light inspection in order to verify if the specimen actually failed after interrupting the assay. This technique is widely used[11,20,23,39] for verifying initial cracks in fragile materials and allows diffuse reflection of a light source to enable visualizing defects and/or cracks. Some samples in which the failure was not easily visible were photographed in a stereomicroscope and then the colors were inverted for negative effect and crack exposure (Fig. 4). After crack identification, each specimen was sectioned and via high resolution microscopy it was possible to observe that the cracks initiated in the resin cement region (Fig. 5). Thus, it was possible to affirm that all ceramic discs failed on the tensile side of the specimen. The software ANSYS used in this study also reported atomically which geometry showed the maximum stress in the setup. For all groups, the ceramic disc intaglio surface was the region of greatest stress value.

Murillo-Gómez et al. (2018)[39] evaluated if both etching protocols affect the microstructural integrity of the lithium disilicate, leucite and hybrid ceramic. The authors found that the self-etching glass-ceramic primer showed lower etching depth than hydrofluoric acid (10%) etching only for the hybrid ceramic. The findings in this study corroborate with the authors, since higher flexural strength for hybrid ceramic was found using the self-etching glass-ceramic primer. These authors found that hydrofluoric acid increased the roughness for all tested ceramics, with the leucite being more affected than the lithium disilicate. Self-etching glass-ceramic primer presented promising results in the present study. This may be related to this material presenting tetrabutylammonium dihydrogen trifluoride as etching agent, which is less aggressive etchant than hydrofluoric acid.[7] The hydrofluoric acid etching eliminates the silica content from the hybrid ceramic surface, producing greater and deeper glassy phase dissolution and increasing the defect population in the material microstructure, probably increasing the risk of crack propagation.

In using the finite element analysis it was possible to observe that the larger the elastic modulus of the ceramic material, the greater the capacity to retain stress while preserving the adjacent structure,[36] which in this case was the resin cement. In that way, the lithium disilicate ceramic material could support higher stress values before fracture. This characteristic corresponds to the high flexural strength values of this material when compared to the other evaluated ceramics.[41,42] In the present study this behavior remains even after the cementation procedure, however, the biaxial flexural strength could be affected depending on the surface treatment and aging.[20] As study limitations it is important to note that factors such pH variation,[44] mechanical cycling[11] and different resinous cement agents[45] are not simulated in the present study and could influence the mechanical response of the ceramic restoration. For that reason, the presented results should be interpreted with caution and complemented with further studies prior to any direct clinical extrapolation.

CONCLUSIONS

The following conclusions can be made based on the results:

Acid etching or self-etching ceramic primer promote similar immediate biaxial flexural strength for each ceramic.

In the long-term, superior strength was observed using acid etching for lithium disilicate and the self-etching ceramic primer for the hybrid ceramic. While, no difference was observed for leucite-based ceramic.

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Table I. Materials used in this study and their respective manufacturers and compositions.

Material	Trademark	Manufacturer	Composition
Hybrid ceramic	Vita Enamic	Vita Zahnfabrik, Bad Säckingen, Germany	58-63% of SiO_2, 20-23% of Al_2O_3, 6-11% of Na_2O, 4-6% of K_2O, 0.5-2% of B_2O_3, <1% of CaO and <1% of TiO_2
Leucite based ceramic	IPS Empress CAD	Ivoclar Vivadent Schaan, Liechtenstein	64.9% SiO_2, 16.25% Al_2O_3, 11.85% K_2O, 5.37% Na_2O, 1.56% CaO
Lithium disilicate	IPS e.max CAD		57%-80% SiO_2, 11%-19% Li_2O, 0%-13% K_2O, 0%-11% P_2O_5, 0%-8% ZrO_2, 0%-8% ZnO, 0%-5% Al_2O_3, 0%-5% MgO
Conventional adhesive system	Multilink Primer A		Water iniciators
	Multilink Primer B		Phosponic acid acrylate, hydroxyethyl methacrylate, mathacrylate modified polyacrylic acid, stabilizer
Dual-cure resin cement	Multilink		Dimethacrylate, HEMA, water, barium glass filler and silicon dioxide filler, initiators, ytterbiumtrifluoride, catalysts and stabilizers, pigments

Table 2. Groups distribution according to the material (lithium disilicate, leucite based ceramic and hybrid ceramic), surface treatment (acid etching or self-etching glass-ceramic primer) and aging (with or without 10,000 thermocycles). And, descriptive statistics (in N) and Tukey test. Similar capital letters represent groups without difference.

Material	Surface treatment	Aging	Strength ± St Dev (MPa)	Tukey test
Hybrid ceramic	Hidrofluoridric acid etching + Silane*	Without	443.9 ± 44	CDE
		With	424.3 ± 48	E
	Self-etching glass-ceramic primer **	Without	470.2 ± 42	ABCD
		With	459.8 ± 68	BCD
Leucite based ceramic	Hidrofluoridric acid etching + Silane*	Without	452.6 ± 36	CDE
		With	436.8 ± 21	DE
	Self-etching glass-ceramic primer **	Without	472.9 ± 38	ABC
		With	439.3 ± 29	CDE
Lithium disilicate	Hidrofluoridric acid etching + Silane*	Without	491.8 ± 47	AB
		With	490.3 ± 19	AB
	Self-etching glass-ceramic primer **	Without	499 ± 17	A
		With	445.3 ± 39	CDE

*The ceramic surface was etched with 10% hidrofluoric acid (Condac porcelana. FGM. Joinville. Brazil) during 60 s for lithium disilicate (e.max CAD. Ivoclar Vivadent. Schaan. Liechtenstein) and 20 s for Leucite based ceramic (Empress CAD. Ivoclar Vivadent. Schaan. Liechtenstein) and Hybrid ceramic (VITA Enamic.VITAZahnfabrik. Bad Säckingen. Germany). Then. the surfaces were rinsed and dried. Monobond Plus was then applied on the surface. and the time for volatilization of the solvent was observed before cementing. ** The ceramic surface received an active application of Self-etching glass ceramic primer (Monobond Etch and Prime. Ivoclar Vivadent. Schaan. Liechtenstein) for 20 seconds. followed by 30 seconds of setting. The samples were then washed with running tap water and dried with an oil-free air jet.

Table 3. Three-way Analysis of Variance results for load to fracture, according to the factors type of ceramic material, surface treatment and aging.

Source	**DF**	**Adj SS**	**Adj MS**	**F-Value**	**P-value**
Material	2	455967	227983	26.48	0.000*
Surface treatment	1	30912	30912	3.59	0.061
Aging	1	255948	255948	29.73	0.000*
Material* Surface treatment	2	214652	107326	12.47	0.000*
Material*Aging	2	14707	7354	0.85	0.428
Surface treatment*Aging	1	52668	52668	6.12	0.015*
Material* Surface treatment*Aging	2	80286	40143	4.66	0.011*
Error	108	929739	8609		
Total	119	2034879			

Figure Legends

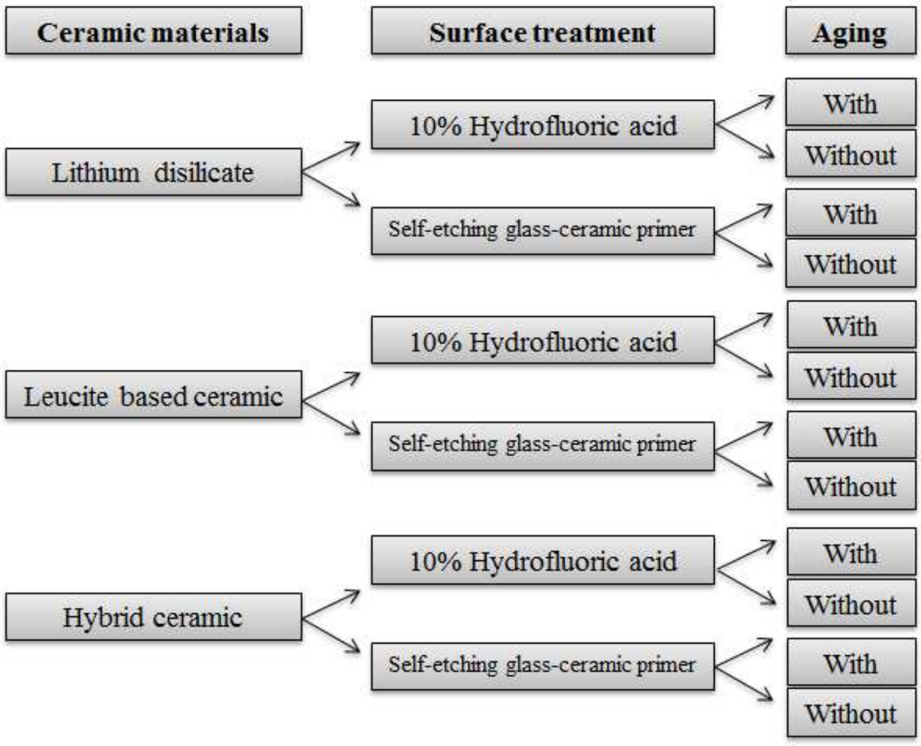

Figure 1. Flowchart of the allocation of experimental groups (n = 10 per group) with 3 experimental factors based on “Ceramic material” - 3 levels, “Surface treatment” - 2 levels and “Aging” - 2 levels.

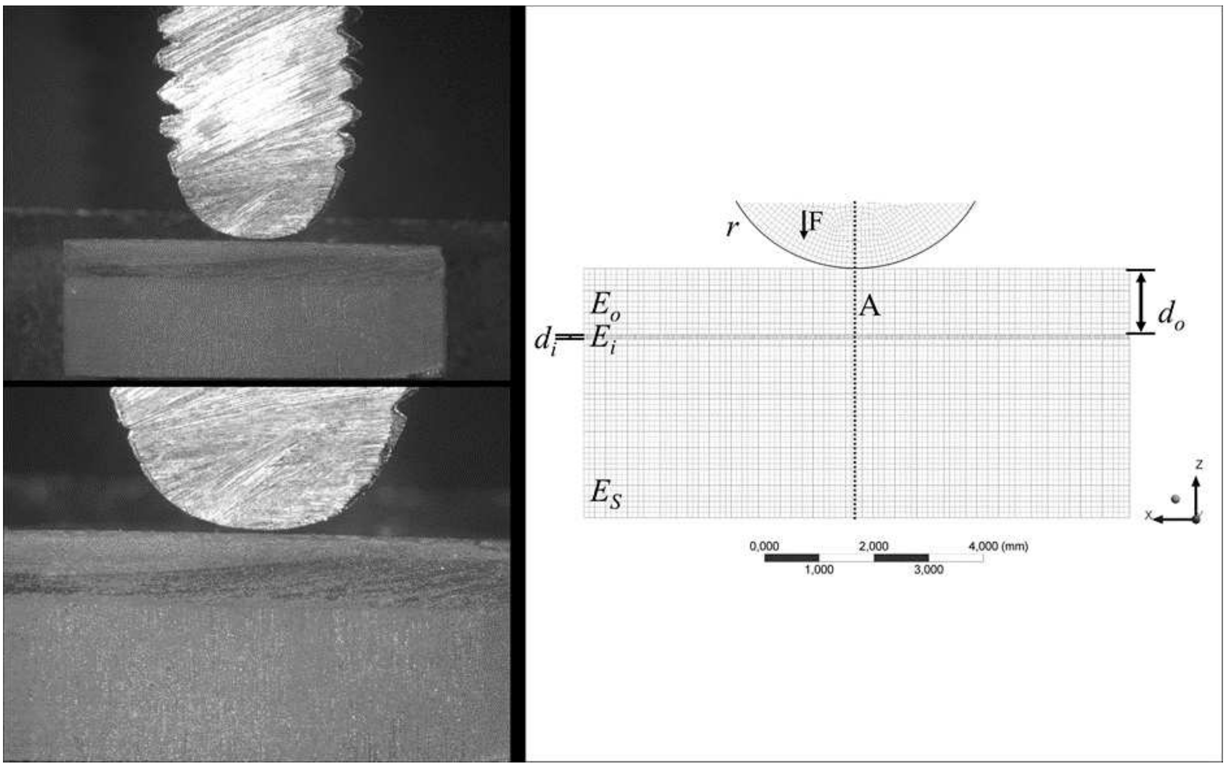

Figure 2. Schematic of tri-layers with brittle outer layer of thickness *do,* and modulus *Eo* and brittle inner layer of thickness *di* and modulus *Ei* on compliant substrate of modulus *Es*, in contact at top surface with sphere of radius *R* at load *F*. A is the symmetric axis during the modeling.

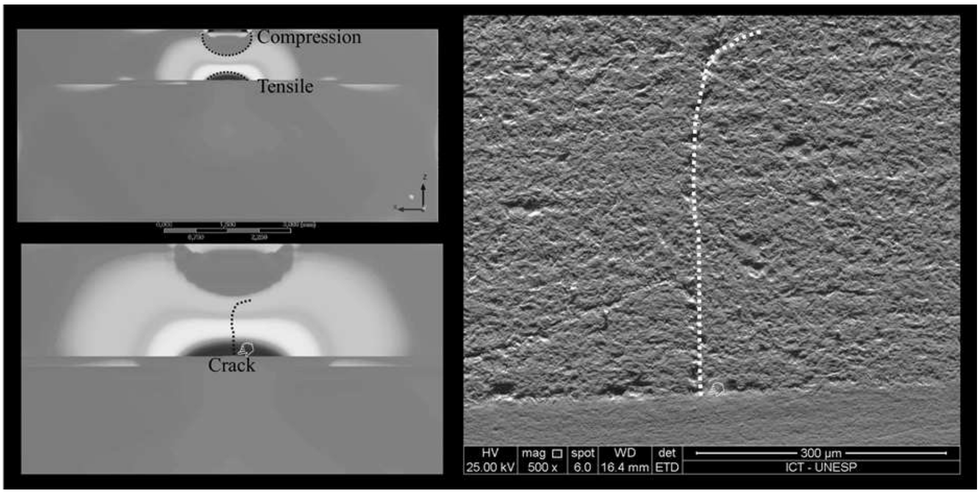

Figure 3. Exemplification of the compression and tensile zones during the numerical calculation and the compression curl in the in vitro sample as a result of these zones.

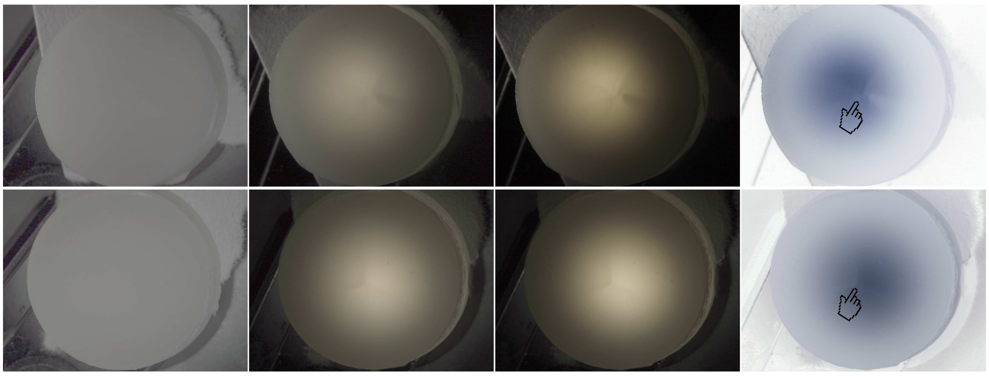

Figure 4. Analysis of representative failed discs using a stereomicroscope (7.5x) under different illuminations. First row without external light incidence, second row with light source under the sample for transilumination approach and crack evidence, third row with the increase of contrast in 50% and the last row the negative image.

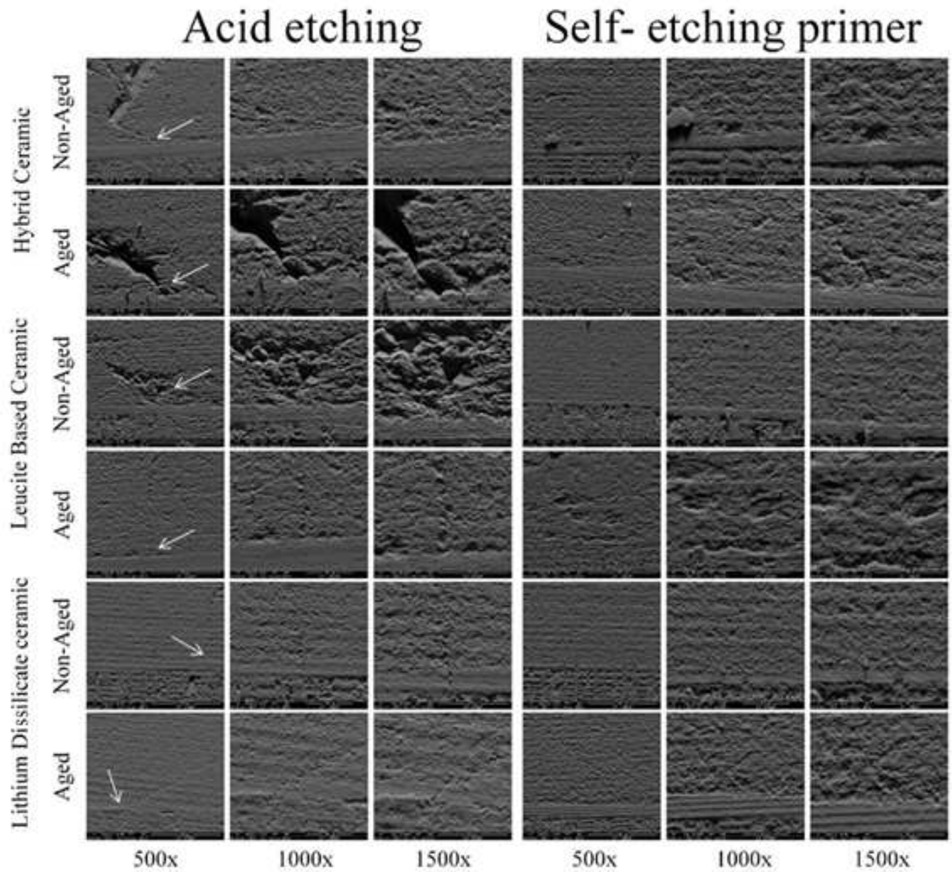

Figure 5. Representative SEM analysis of failed discs under different magnifications (500×, 1000× and 1500×). The arrows and pointers indicate the failure origin.

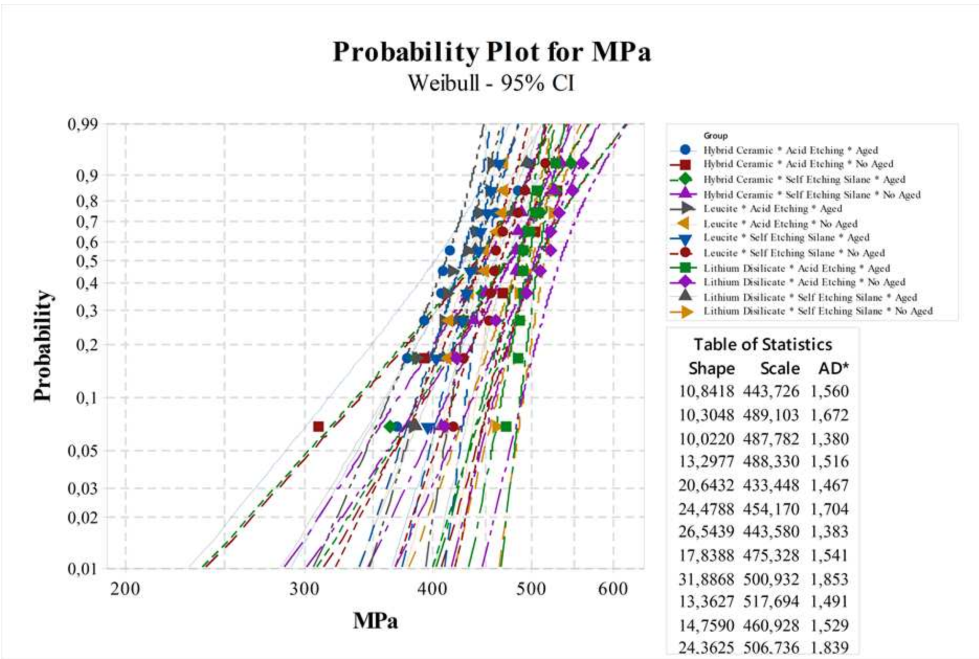

Figure 6. Weibull plot for biaxial flexural strength data according to the ceramic*surface treatment*aging interaction. This probability plot shows the Weibull modulus (Shape) and dispersion of strength values (Scale).